

# SCIENCE FOR CERAMIC PRODUCTION

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## PARTICULARS OF POLYCRYSTALLINE OXIDE CERAMIC CREEP AT TEMPERATURES TO 1600°C

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The results of an investigation of creep in a polycrystalline metal-oxide ceramic at temperatures to 1600°C and under high loads are presented. These temperatures lie near the transition from the brittle to plastic behavior of the ceramic. The loads are close to the ultimate strength of the materials at the given temperature. It is shown that the elementary mechanism of deformation in the entire plasticity range of the samples is diffusion-viscous (vacancy or interstitial) flow in its different forms — diffusion motion of dislocations and crystal boundaries. The data obtained are in agreement with the supposition that for a transition from brittle to plastic fracture of a ceramic a critical vacancy concentration in the ceramic is required. These vacancies are formed as a result of thermal activation and determine the deformation of the materials.

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**Key words:** polycrystalline oxide ceramic, creep, brittle–plastic transition.

The results of systematic studies of creep of metal-oxide polycrystalline ceramics are presented in [1, 2]. The experiments were performed in the region of variation of the experimental conditions where the samples show appreciable irreversible flow deformation — plasticity<sup>4</sup> — at comparatively high temperatures (> 1600°C) and under low loads (to 3 MPa). It was shown that under such conditions the main deformation mechanism at the atomic level is the diffusion of atoms (ions) along vacant sites of the crystal lattice or interstices with all its variety — the movement of dislocation or crystal boundaries along vacancies.

The works cited also note the main features of the behavior of the samples — practically only stationary creep (i.e., with constant rate) is observed; as stress increases, the rate of

the process increases as a power law with exponent  $n$  about 1 or slightly higher (to 2 – 2.5); as temperature increases the creep rate increases exponentially, and the activation energy of the process corresponds to the activation energy of self-diffusion. Exceptions from this regular behavior occurred only in measurements of the structure of the materials in the course of the tests or with a change of the deformation mechanism. The studies indicated above were continued for the behavior of the experimental materials at lower temperatures and under higher loads, close to the ultimate strength of the samples [3]. In the opinion of the authors, such experiments are of great interest from the standpoints of applications as well as theory.

The aim of the present article is to present experimental results on the determination of creep in an air atmosphere of polycrystalline oxide ceramic in the temperature interval 1450 – 1550°C and under loads 5 – 60 MPa.

The investigations were performed at temperatures and under loads for which the measured rate of deformation (strain) was in the range  $10^{-1}$  –  $10^{-5}$  h<sup>-1</sup>. This was convenient for a laboratory study, because the longest duration of an individual determination did not exceed 100 h with motion sensor sensitivity of about 1 μm. At the lower limit (1400°C) of the experimental temperature range (i.e., for measured strain rates of the order of  $10^{-4}$  –  $10^{-5}$  h<sup>-1</sup> with the sensor operating at the threshold of sensitivity) reliable

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<sup>4</sup> The term “plasticity” is used somewhat arbitrarily, since plastic deformation of a ceramic occurs by diffusion and by means of viscous flow of a glassy phase. The plasticity of inorganic materials in general also includes mechanisms of plastic deformation that are inherent to metal bodies — slip of dislocations and block as well as crystal boundaries and even individual sections of a body as a whole.

**TABLE 1.** Some Characteristics of the Experimental Samples

Mass index	Ceramic type	Predominant crystal size, $\mu\text{m}$	Apparent density, $\text{g}/\text{cm}^3$	True porosity, %	Bending strength, MPa, at temperature, $^{\circ}\text{C}$				
					20	1400	1450	1500	1550
K-35	$\text{Al}_2\text{O}_3$	35	3.90	2.5	125	60	54	51	42
ShP-25	$\text{MgAl}_2\text{O}_4$	25	3.50	2.2	120	30	20	15	13
P-12	$\text{MgO}$	12	3.47	3.0	120	65	52	45	40
P-25	$\text{MgO}$	25	3.50	2.2	105	60	73	62	55

results were not always obtained because of noise from external mechanical signals. Under such conditions weak strain accumulates very slowly and the process is very prolonged (the nonstationary period can continue to 30 h and longer). For this reason, in the opinion of the authors, the results can only be regarded as qualitative. We also note that the strain rate as well as the strain magnitude and time to fracture were determined for small sample sizes — about five parallel samples.

Materials based on periclase, alumina-magnesia spinel, and corundum and quite often used in high-temperature engineering were also included in the tests (see also [3]). To study the effects of crystal size, two modifications of periclase, differing by the predominant crystal size — 12 and 25  $\mu\text{m}$ , were included, while spinel and corundum were represented by a single modification — 25 and 35  $\mu\text{m}$  (according to optical microscopy), respectively. The characteristics of the samples [3] are presented in Table 1.

The temperatures chosen correspond to a transition from brittle to plastic fracture of the samples [4] but closer to the plastic behavior range (as is well known, flow deformation is not observed during brittle fracture [5]). According to existing ideas, the brittle–plastic transition temperature is largely determined by the crystal-chemical particulars and structure of the materials. As a rule, the role of a compactly sintered ceramic is quantitatively evaluated according to the size of its crystals.

Ordinarily, this transition is interpreted as a phase transition with a change of the chemical-bond type, specifically, metallization of the ionic-covalent component of the bond [6]. Such transformations are accompanied by a considerable change of the vacancy concentration, so that it is supposed that for macroscopic plasticity of brittle bodies thermodynamic conditions for the formation of a critical density of point defects must be created.

The particulars of deformation and fracture in the region of this transition under prolonged loading of a polycrystalline ceramic under a constant mechanical load are presented in [3]. The present article examines the behavior of these materials in regard to their creep. Investigations have shown that of the three types of ceramics studied periclase is most plastic and corundum least plastic under the test conditions; spinel occupies an intermediate position. It can be supposed that the temperature 1400 $^{\circ}\text{C}$  lies below this transition for

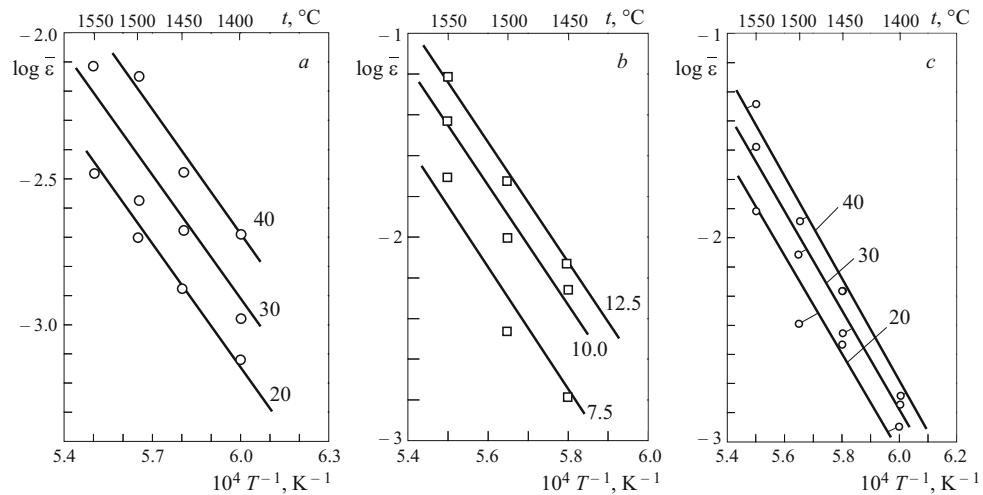
periclase, while for corundum it corresponds to the onset of the transition.

It was also shown in [3] that the curves of creep to fracture at temperatures below 1600 $^{\circ}\text{C}$  had, as a rule, two sections — nonstationary and stationary (with constant rate) deformation; sometimes a third section of accelerated creep prior to fracture was observed. Usually, the nonstationary stage is ascribed to transient processes of structural change in the samples at the time of loading and redistribution of the existing strains in them [7]. At high test temperatures transient processes are fast and the deformation which they determine enters into the initial, instantaneous change of the dimensions of the sample. At low temperatures they are slow and manifest as nonstationary creep. In addition, deformations arising during annealing of “quenched” nonequilibrium structural defects as well as during the relaxation of stresses at concentrators make their own contribution. All these nonequilibrium states are formed when samples are cooled from high temperatures in the course of conventional production of ceramics by sintering.

In the present work it was not possible to ascertain the explicit dependence of the duration of the nonstationary period and the magnitude of the deformation measured on the stress and temperature of the tests. As noted in [3], the differences in the behavior of individual samples are influential here. However, the general regularities in the change of the behavior of the materials with a change in temperature or load were established on the basis of an analysis of extensive experimental data. They reduce essentially to the following manifestations which sometimes also occur during tests performed on metals.

For all experimental materials the nonstationary period is observed at the low temperature limit of the tests (1400 $^{\circ}\text{C}$ ), as well as with an increase of temperature under loads close to the ultimate strength. The duration of this period decreases with increasing temperature and decreasing external force. It is manifested most weakly in periclase and most strongly in corundum. For periclase samples, the deformation during the nonstationary period is small, while for corundum it is very noticeable. In most cases, spinel falls in an intermediate position but explicit dependences could not be established.

Better determined regularities were obtained by studying the change of the rate of stationary creep as a function of the test conditions (temperature, load) for each type of ceramic. The experimental data are presented in the form of a piece-



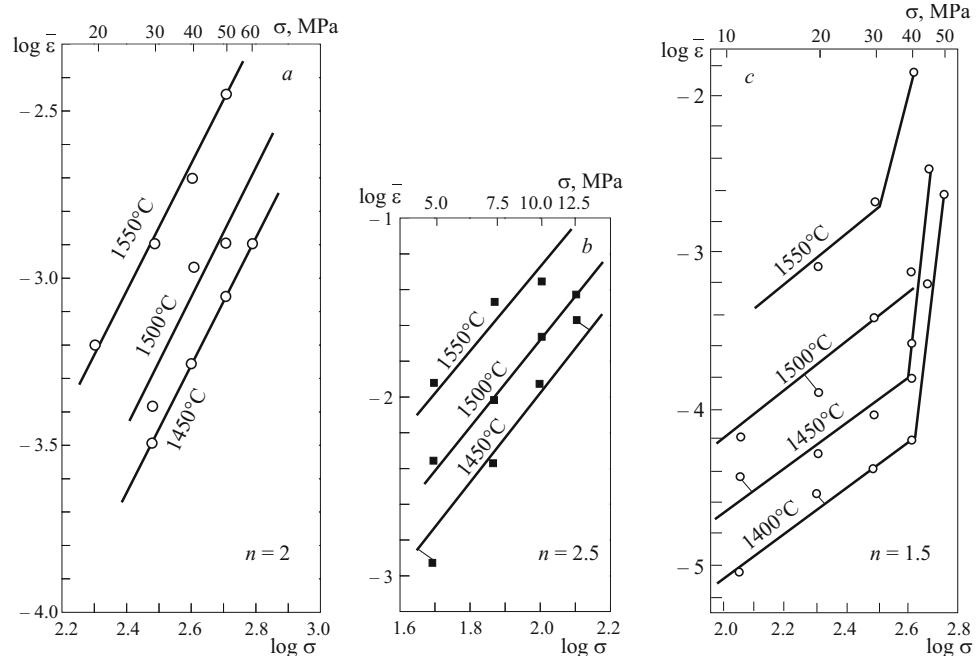
**Fig. 1.** Temperature dependence of the rate of stationary creep of the samples (see Table 1): a) P-25; b) Sh-25; c) K-35 (the load in MPa is indicated on the straight lines).

wise-linear fit in logarithmic and semilogarithmic coordinates, which, in our opinion, simplifies their analysis. Practically all these dependences in the quite narrow experimental range of variation of the variables (temperature and load) are curvilinear and with inflections in terms of the natural quantities.

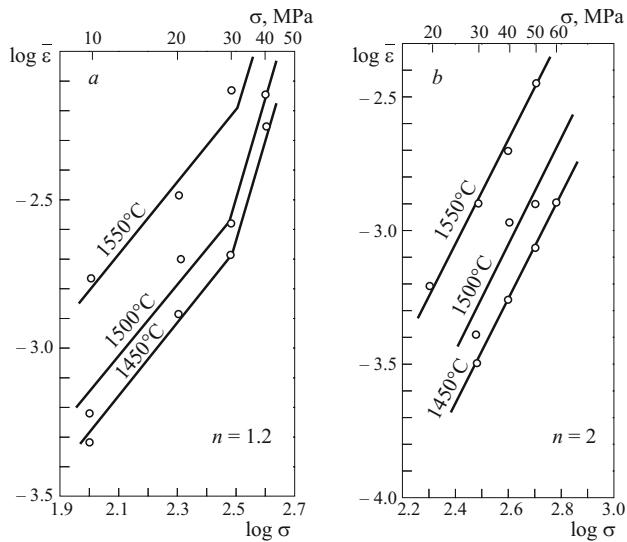
The temperature dependence of the rate of stationary creep of the experimental materials is displayed in Fig. 1. As one can see from the figure, all plots are practically linear in semilogarithmic coordinates, i.e., the function is exponential, just as at high temperatures [1, 2]. The activation energies of the process as calculated from the slopes of the straight lines

are about 400, 600 and 600 kJ/mole for periclase, spinel, and corundum, respectively. These values are practically identical to those determined previously for high temperatures and correlate with the activation energy of self-diffusion in the corresponding compounds. Thus, the results of the studies performed in the present work confirm the conclusion drawn in [1, 2] that the deformation of polycrystals of ceramic materials is of a diffusion nature in the entire temperature range of their plastic behavior.

The data on the rate of stationary creep as a function of the stress (Fig. 2) confirm this result: the plots for all experi-



**Fig. 2.** Rate of stationary creep of samples (see Table 1) versus the load  $\sigma$ : a) P-25; b) Sh-25; c) K-35 (the temperature in °C is indicated on the straight lines).



**Fig. 3.** Rate of stationary creep of the samples (see Table 1) versus the load  $\sigma$ : a) P-12; b) P-25 (the temperature in °C is indicated on the straight lines).

mental materials are power-law functions with exponent 1 or somewhat higher (to 2.5).

The deviations from the theoretical variant of purely vacancy creep are completely explainable. The presence of nonstationary creep can be due to the presence of nonequilibrium stress concentrators and defects. The inflections in the rectilinear logarithmic power-law dependences of the creep rate versus strain in the case of loads preceding fracture are most likely due to the onset of other vacancy mechanisms, such as dislocation climb and vacancy slip of boundaries.

As an example, measurements of the rate of stationary creep of periclase samples differing by the predominant crystal size are presented in Fig. 3. It is evident that as the structure changes the general character of the dependences re-

mains the same, but the absolute magnitude of the rate of stationary creep decreases as crystal size increases (as follows from the diffusion model).

## CONCLUSIONS

The studies performed in this work have confirmed that creep in a metal-oxide polycrystalline ceramic proceeds by diffusion mechanisms in the entire temperature interval of its plastic behavior. On heating, the transition of the brittle state into the plastic state at the macro level is due to the formation of a critical density of point defects in the ceramic. In the case studied here the formation of defects could be due to thermal activation, where as temperature increases, the concentration of defects grows exponentially, i.e., at first slowly and then, after a certain temperature, very rapidly.

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